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14. ABSTRACT

Hybrid laminated composites such as titanium-graphite (TiGr) laminates are an emerging class of structural materials with the potential to enable a new generation of efficient, high-performance aerospace structures. By combining the property sets of dissimilar materials, a synergistic set of properties can be realized. Critical to performance and reliability is the durability of the adhesive interphases between the dissimilar layers. Our program has developed new and superior ways of making highly effective bonds of the metal oxide/epoxy interfaces in such hybrid laminates that are critical to their performance. We achieved this by developing a mixed metal/epoxysilane sol-gel hybrid coupling layer. These hybird materials exhibit excellent properties but there was a deficiency in understanding the fundamental mechanisms behind their performance and reliability. To this end our program also sought to provide the fundamental characterization of the thermomechanical properties of the hybrid layers and the interphase region using a number of new and unique thin-film mechanical and fracture characterization methods.

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Final Report

from

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to

AFOSR/NA
Department of the Air Force

875 North Randolph Street
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Attention: Dr. Joycelyn Harrison
Program Manager

on

Interphase Thermomechanical Reliability and Optimization for

High-Performance Ti Metal Laminates

Principal Investigator:

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AFOSR Grant No. FA9550-08-1-0015

December 2011

STANFORD UNIVERSITY

This is the final report on our program entitled: Interphase Thermomechanical Reliability and Optimization for High-Performance Ti Metal Laminates. The report summarizes progress over the course of the program.

PROGRAM SUMMARY

Hybrid laminated composites such as titanium-graphite (TiGr) laminates are an emerging class of structural materials with the potential to enable a new generation of efficient, high-performance aerospace structures. By combining the property sets of dissimilar materials, a synergistic set of properties can be realized. Critical to performance and reliability is the durability of the adhesive interphases between the dissimilar layers. Our program has developed new and superior ways of making highly effective bonds of the metal oxide/epoxy interfaces in such hybrid laminates that are critical to their performance. We achieved this by developing a mixed metal/epoxysilane sol-gel hybrid coupling layer. These hybird materials exhibit excellent properties but there was a deficiency in understanding the fundamental mechanisms behind their performance and reliability. To this end our program also sought to provide the fundamental characterization of the thermomechanical properties of the hybrid layers and the interphase region using a number of new and unique thin-film mechanical and fracture characterization methods. We developed physics and chemical reaction rate models together with computational methods to model the hybrid molecular structure and resulting mechanical and fracture properties. The program supported 4 doctoral students, two have graduated with PhD's and two are still in progress. The program resulted in a number of technical publications and invited and contributed talks at international conferences. The knowledge developed in the program offers the possibility to engineer, at the molecular level, the hybrid layers in laminated composites for improved thermomechanical reliability.

Objective

Characterize and innovate state-of-the-art titanium graphite hybrid laminates to dramatically enhance material performance and high-temperature capabilities for aerospace systems. Develop a fundamental materials and mechanics basis from which new hybrid laminates and bonded structures can be designed with optimized interphase regions.

Approach

We employed a multiscale approach using novel thin-film and conventional fracture mechanics techniques to probe the hierarchical structure of high-performance titanium graphite and other hybrid laminates. We develop new toughened interphase adhesives for improved bonding to carbon fiber prepregs. We developed and exploited a novel high-productivity (combinatorial) thin-film fabrication capability to rapidly screen and optimize hybrid organic-inorganic interphase molecular architectures between a range of metal-oxide and adhesive layers. By changing the precursors, solvents, and process variables, a wide range of molecular structures was realized which facilitated an expanded design space for high-performance hybrid laminates in general. Studies were expanded to include integrating other metal foils and substrates such as Ti- and Zr-based metallic glasses, Al-alloys, silicon wafers, and even Indium Tin Oxide (ITO) conducting transparent electrodes. The work with silicon and ITO substrates was intended to provide the basis for integrating embed stretchable-silicon microsenor networks for damage and intelligent performance monitoring without introducing/creating weak regions for thermomechanical damage initiation. Full environmental and temperature controlled thin-film adhesion and cohesion testing capabilities were employed to identify and characterize the principal mechanisms of adhesion and damage in a variety of aggressive test environments, applied loading mode mixities, and complex fatigue loading conditions. We developed models of the underlying failure mechanisms using thermomechanical and chemical reaction rate models to facilitate reliability predictions.

Scientific Challenge

State-of-the-art titanium graphite hybrid laminates involve a complex hierarchical layered structure engineered over a wide range of length scales. The interphase region between the principal titanium and reinforced polymer layers contains critical films and interfaces that determine load transfer, environment and temperature sensitivity, and ultimate thermomechanical performance. Surprisingly, while the predominant damage and fracture processes in laminates nearly always involve delamination of such constituent layers as precursors to macroscopic damage, virtually nothing is known regarding the fundamental degradation processes that lead to loss of adhesion, debonding of interfaces, and cohesive cracking of vital thin films in the interphase regions of the laminate structure. The fundamental scientific challenge was therefore to employ a hierarchical approach involving a combination of novel thin-film and conventional techniques to characterize and model the fundamental processes of adhesion and damage evolution in the interphase region, to determine the effects of interphase layer composition and molecular structure, to determine the role of service environment including moisture and chemical species, and finally the effects of temperature and loading type on the debond path and its time- or loading-cycle dependence. We want to develop a fundamental materials and mechanics basis from which new interphase regions can be innovated for novel hybrid laminates. The research will have direct implications for the reliability of other high-performance structural adhesive joints, for a variety of functional coatings, and for embedding microsensor networks.

PROGRESS

Annual Accomplishment Summary: We have continued to focus our efforts to develop a fundamental materials and mechanics basis on which new hybrid materials can be designed with optimized composition and molecular structure (**Fig. 1**). As before, we have used a multiscale approach combining thin-film and conventional processing and fracture mechanics techniques to fabricate and probe the hierarchical structure of hybrid materials and the toughening mechanisms of novel second phase toughened epoxy resins. Specifically, we have exploited novel high-productivity thin-film microelectronic-based fabrication capabilities we recently developed to rapidly screen and optimize hybrid organic-inorganic interphase molecular architectures between a range of metal-oxide and adhesive layers. By changing the precursors, solvents, and process variables, a wide range of molecular structures can be realized which has facilitated an expanded design space for high-performance hybrid laminates.

Leveraging from fundamental knowledge built through experimental optimization of our ZrO_x/epoxysilane hybrid films and computational molecular models, we have begun to explore the development of nanoporous hybrids using a sacrificial porogen approach which will provide enhanced property sets for ultra-lightweight applications.

We have also continued our collaboration with Applied Poleramics in the development of novel second phase toughened resins to improve the performance of thin bondline structures. Complex fracture mode and environmental durability assessment of the adhesive bondlines has been conducted to better understand the kinetics of reaction and its impact on long-term reliability. Studies probing the influence of temperature and humidity on subcritical debonding have been conducted, and efforts to understand fatigue behavior are currently underway.

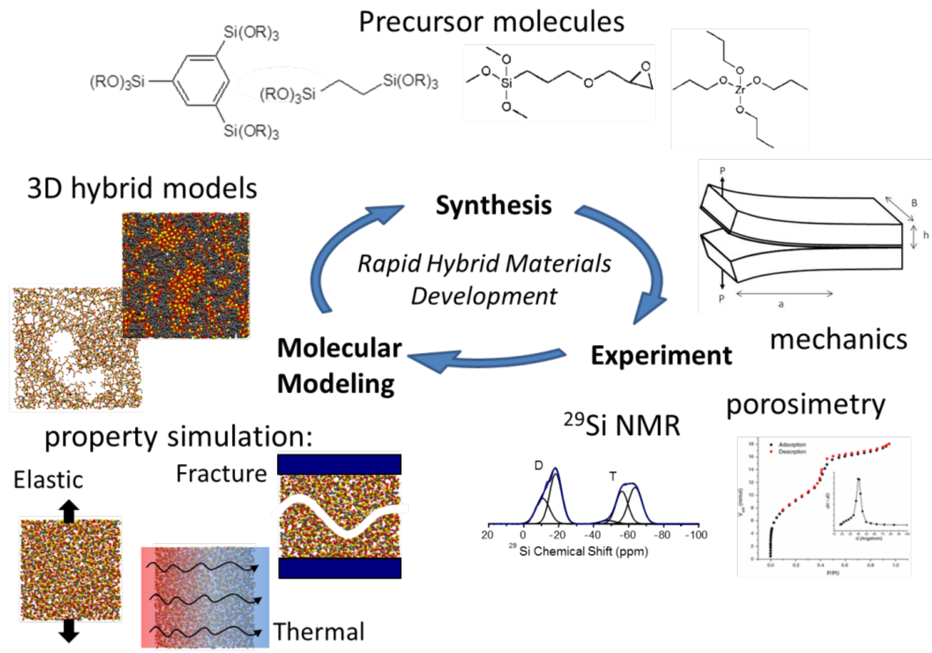

Figure 1. Schematic illustrating the iterative development process of hybrid materials and the interconnection of hybrid synthesis, experimental characterization and molecular simulation.

The following sections briefly describe progress in a number of areas for the fourth year of our AFOSR program.

<u>Significant Accomplishment 1:</u> Synthesis and Processing of Nanoporous Molecular Hybrids

Our previous AFOSR work has demonstrated the efficacy of ZrO_X/epoxysilane hybrid films in strengthening the adhesive interphase region between dissimilar materials in fiber metal laminate structures. We believe certain property sets, including optical, dielectric and thermal, can be further enhanced with the addition of nanoporosity to create novel ultra-lightweight and multi-functional hybrid materials. In doing so, it will be critical to retain the robust mechanical and fracture properties obtained in our optimized dense hybrid films.

Several methods of nanopore development have been investigated for these complex hybrid structures including the porogen burnout approach which results in an amorphous morphology, as well as a nanoparticle template approach which results in a spherical morphology (**Fig. 2**). To synthesize nanoporous molecular hybrids, we have employed the porogen burnout approach using a commercially available sacrificial porogen, polyoxyethylene(23)laurylether. Using a nucleation and growth process with subsequent decomposition and volitization of the porogen in high temperature cure environments, we have begun to develop ultra-lightweight versions of our ZrO_X/epoxysilane hybrid. These experimental techniques will be coupled with additional molecular modeling to investigate the relaxation effects that may occur following porogen burnout to prevent the possibility of pore collapse.

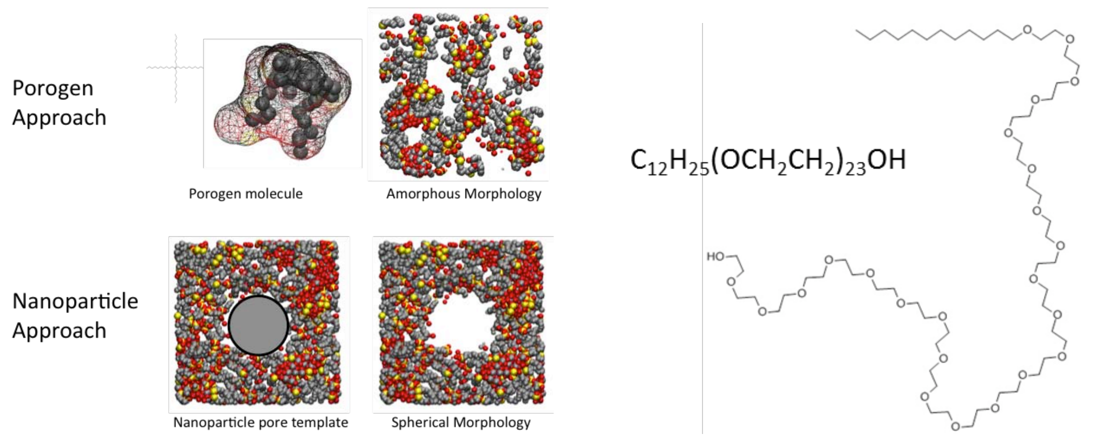

Figure 2. Two approaches can be applied in MD simulations to introduce nanoporosity into hybrid structures. The porogen burnout approach results in a random amorphous morphology that can accurately mimic realistic structures. The nanoparticle template approach can be used to develop morphologies with strictly spherical inclusions, which is critical for interfacial studies and nanopatterned structures. On the right, a schematic of the polyoxyethylene(23)laurylether porogen molecule which was used as the sacrificial porogen during hybrid film synthesis.

The weight percentage of porogen was varied in the sol-gel solution during the synthesis of our hybrid films, with an estimated 30% porosity corresponding to ~25 wt% porogen additives based on techniques described in literature. Early measures of fracture energy have indicated a trend of decreasing adhesive strength with increasing porogen or film porosity (**Fig. 3**). At this stage, it is unclear whether or not porosity has been successfully introduced into the hybrid structures through porogen burnout during the cure cycle, or if the porogen molecule is influencing the reaction kinetics of the sol-gel synthesis process. Nanopore characterization using ellipsometry, porosimetry, x-ray reflectivity (XRR) and scanning electron microscopy (SEM) will be conducted to fully characterize the structures of the resultant films. Upon successful synthesis of the nanoporous hybrids, we will systematically control pore size distribution and volume fraction to explore their influence on mechanical (elastic stiffness, strength, hardness) and fracture properties (adhesive, cohesive failure) as well as environmental degradation and diffusion mechanisms.

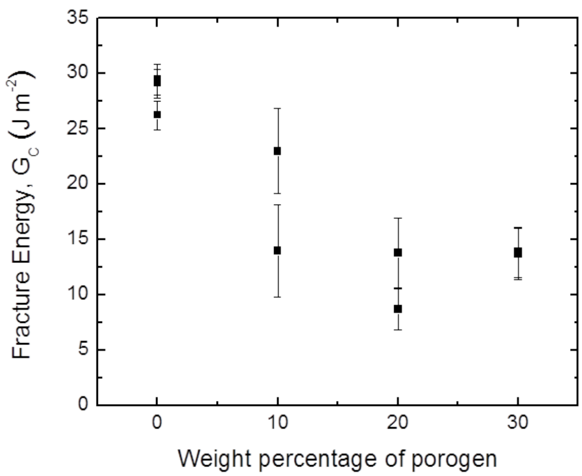

Figure 3. Critical fracture energy of ZrO_X/epoxysilane hybrids as a function of weight percent porogen additions, where 30% porosity is estimated to correspond to ~25 wt% porogen additives.

<u>Significant Accomplishment 2:</u> Development and Fracture Characterization of Second Phase Toughened Adhesives for Fiber Metal Laminates

We have continued our collaboration with Applied Poleramics to develop and characterize a new series of second phase toughened adhesives for enhanced performance and reliability of thin bondline structures. Several studies have been employed to assess the fracture characteristics of toughening strategies including second phase rubbers, rubber particle sizes, silica nanoparticles, hard core shells and epoxy cross-linking agents relative to a baseline single phase Bis-A toughened epoxy resin.

Double cantilever beam specimens were fabricated using spray-coating techniques and pressurized cure cycles to form 20μm thick bondlines between Ti substrates. Critical fracture energy measurements for the baseline resin and six toughened resin structures are showin in **Fig. 4**. In addition to adhesion to bare titanium, specimens with an additional ZrO_x/epoxysilane hybrid sol-gel film were also fabricated to measure the infuence of the interphase layer on adhesion. Initial critical fracture energy measurements have indicated significant improvements from MOD2 which is toughened with 0.4μm butadiene / acrylic hard core shells. The figure also illustrates the importance of the organic-inorganic sol-gel film (denoted by SG) in improving the adhesion between the toughened resins and the underlying Ti substrate. In the absence of the sol-gel film, many of the specimens failed adhesively at the Ti/resin interface. However, with the graded hybrid layer, failure was observed to be cohesive within the toughened resin at fracture energies up to three times greater than adhesive debonding measurements. Scanning electron micrographs of the fracture surfaces of select epoxy resins are shown in **Fig. 5**, revealing a nanoporous channeled structure with diameters on the order of 100-250nm in the baseline resin matrix. This porous structure may explain the lack of toughening observed in resins with second phase inclusions on the order of the pore diameters, including MOD4 which was toughened with ~50-100nm silica nanoparticles.

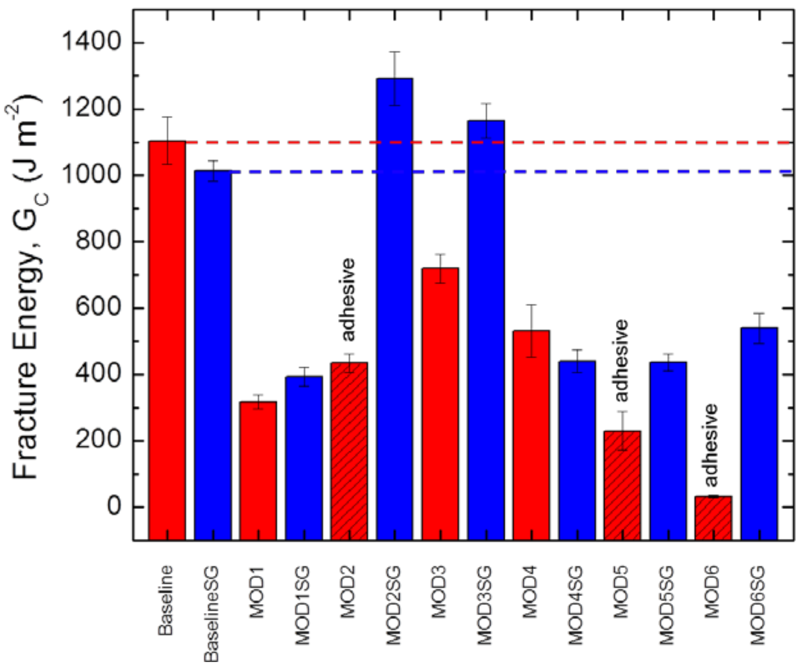

Figure 4. Critical fracture energy measurements for the baseline single phase Bis-A resin compared to second phase toughened formulations. Comparison between resins deposited with and without the hybrid sol-gel film indicates significant improvements in fracture strength with the addition of the interphase layer. The MOD2 formulation outperformed the baseline resin with the addition of hard core shell particles.

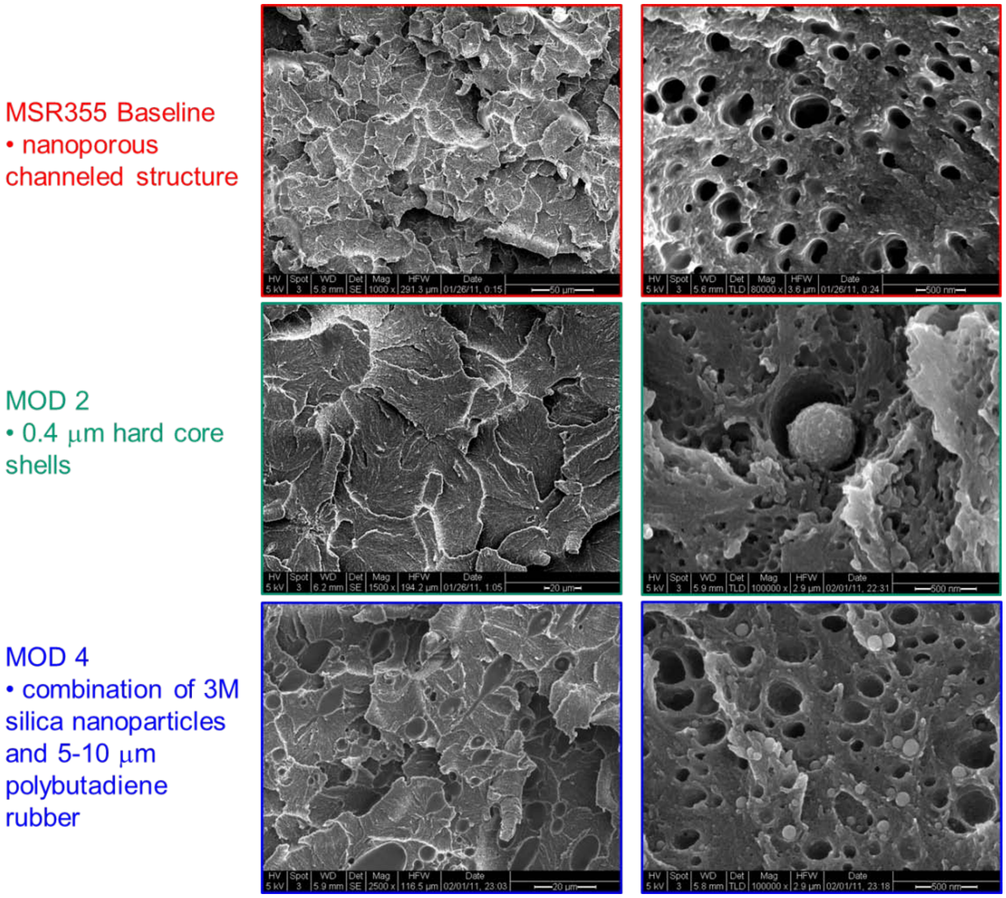

Figure 5. SEM fractographs reveal a nanoporous, channeled structure in the baseline Bis-A resin.

Significant Accomplishment 3: Characterization of Environmentally-Assisted Debond Kinetics and Fatigue Crack Growth in Second Phase Toughened Adhesives

In addition to characterizing the cohesive and adhesive fracture processes, it is also of critical importance to investigate environmentally assisted crack growth kinetics that cause in-service degradation. To evaluate the role of service environment on the degradation of the toughened resins and hybrid structures, subcritical debonding experiments were conducted in environments with varying temperature and humidity levels.

Fig. 6 illustrates crack growth rates as a function of applied strain energy release rate for the baseline (single phase Bis-A resin), MOD2 (0.4μm butadiene / acrylic hard core shells) and MOD4 (silica nanoparticles and 5-10μm rubber particles) resins with the underlying sol-gel interphase layer. In this study, temperature was held constant at 20°C while relative humidity was varied systematically from N_2 (no humidity) to 50%RH and 80%RH. All subcritical debonding experimentas resulted in cohesive failure within the epoxy resin demonstrating the stability and strength of the hybrid layer in moist environments. Both the baseline and MOD2 resins experienced increased crack growth rates with increasing relative humidity, demonstrating susceptiblity to moisture assisted debonding. However crack growth in MOD2 is shown to occur at significantly higher G values, consistent with the critical fracture energy measurements described earlier. In contrast, the crack growth rates measured for MOD4 show no clear sign of moisture sensitivity. This insensitivity may result from reduced or inhibited moisture transport through the porous channeled structure due to blockage by the silica nanoparticles. These observed trends can be used to develop adhesives that not only demonstrate superior fracture energy, but also effectively suppress moisture-assisted debonding.

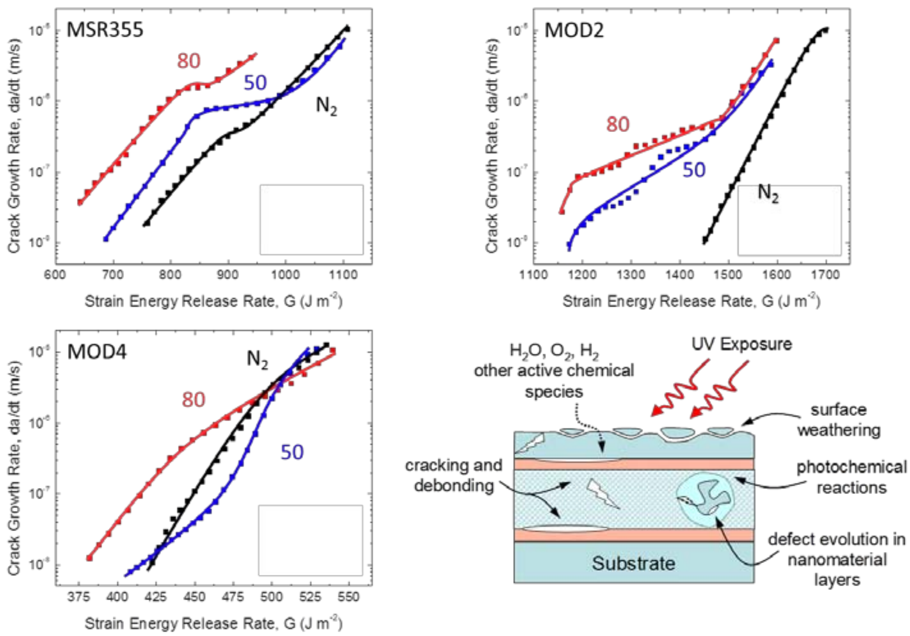

Figure 6. Subcritical debonding of second phase toughened resins in 20°C environments with varying humidity levels from N_2 to 50%RH and 80%RH.

The influence of temperature on crack growth kinetics is shown in **Fig. 7**. Relative humidity was held constant at 50%RH and the temperature was variable at 20°C, 80°C and subzero temperatures of -20°C. The influence of elevated temperatures resulted in the crack growth rate curve shifting to lower values of strain energy release rate, indicating significantly accelerated crack growth rates for a given G. It also shows significantly depressed threshold values, below which crack propagation will not occur. In contrast, the change between room temperature and subzero environments showed little influence on the measured debond growth rates.

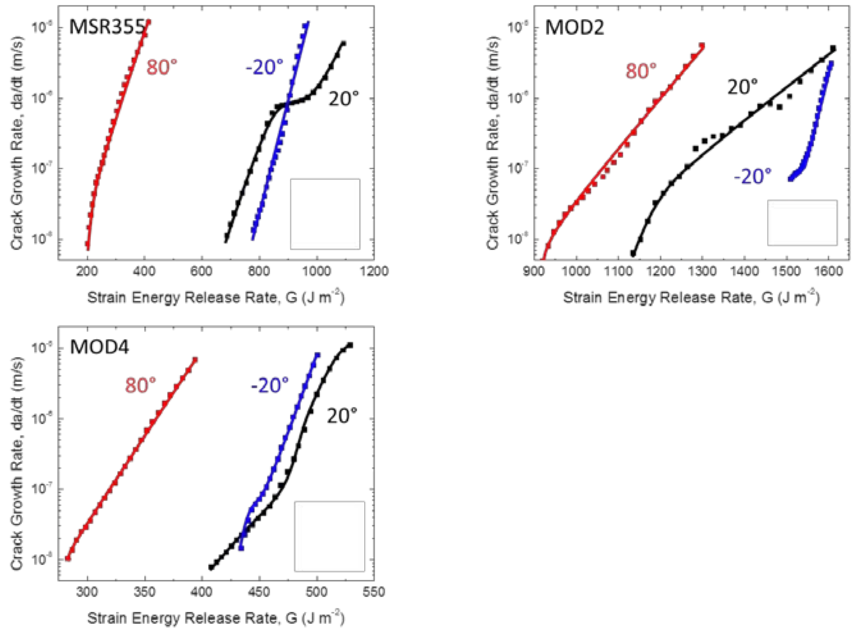

Figure 7. Subcritical debonding of second phase toughened resins in 50%RH environments with varying temperature from -20°C to 20°C and 80°C.

In addition to environmental stresses, we have also begun to characterize the influence of complex loading on degradation of these toughened resins. Preliminary experiments have been conducted to characterize crack growth behavior of the toughened resins under fatigue loading conditions (**Fig. 8**). Using the double cantilever beam fracture mechanics test geometry, a cohesive fatigue crack was driven through the center of the toughened epoxy layer. Cyclic loading was found to significantly accelerate crack growth rates compared to static fatigue behavior, demonstrating the importance of mechanical fatigue phenomena on the reliability of these toughened resin networks. Work is currently underway to characterize fatigue phenomena over a range of cyclic frequencies and applied load ratios.

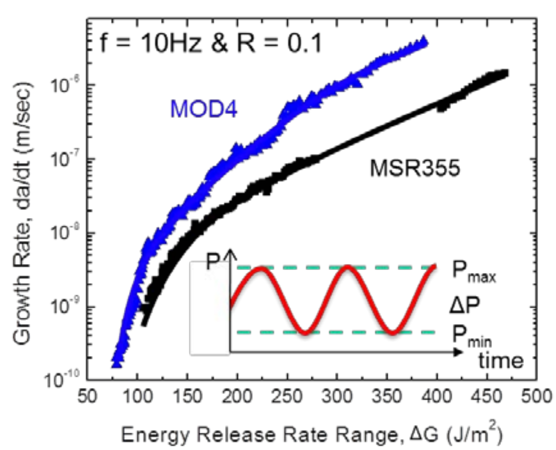

Figure 8. Preliminary fatigue debonding characterization of baseline MSR355 Bis-A resin and silica nanoparticle & second phase rubber toughened MOD4.

Acknowledgement/Disclaimer

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PERSONNEL SUPPORTED ON AFOSR PROGRAM

Mark Oliver	(Ph.D. graduated)	Graduate Student Research Assistantship
Anay Kamer	(Ph.D. graduates)	Graduate Student Research Assistantship
Jeffrey Yang	(Ph.D. current)	Graduate Student Research Assistantship
Linying Wang	(Ph.D. current)	Graduate Student Research Assistantship

Prof. Reinhold H. Dauskardt Professor (PI)

PUBLICATIONS

M.S. Oliver, K.Y. Blohowiak, R.H. Dauksardt, "Molecular Structure and Fracture Properties of ZrO_X/Epoxysilane Hybrid Films" *Journal of Sol-Gel Science and Technology* 55, 360, 2010.

M.S. Oliver, R.H. Dauskardt, "Mechanical Fatigue of Hybrid Glasses" *Small* 6, 1892, 2010.

M.S. Oliver, G. Dubois, M. Sherwood, D.M. Gage, R.H. Dauskardt, "Molecular Origins of the Mechanical Behavior of Hybrid Glasses" *Advanced Functional Materials* 20, 2884, 2010.

J. Yang, M.S. Oliver, R.H. Dauskardt, "Optimized Adhesive Bonding to Bulk Metallic Glass Substrates" SAMPE 2010, Seattle, WA, 2010.

M.S. Oliver, R.H. Dauskardt, "Crack Growth Mechanisms in Sol-Gel Adhesive Coupling Layers" SAMPE 2010, Seattle, WA, 2010.

Mark S. Oliver, Kay Y. Blohowiak, Reinhold H. Dauskardt, "Reliability of Adhesive Interphases for Titanium-Graphite Laminates," SAMPE 2008, Los Angeles, CA, 2008.

J. Yang, M.S. Oliver, R.H. Dauskardt, "Influence of Substrate Isoelectric Point on Hybrid Organic-Inorganic Sol-Gel Adhesion", *Advanced Functional Materials, in review.*

J. Yang, M.S. Oliver, R.H. Dauskardt, "High Performance Bonding to Metallic Glasses Using Hybrid Organic-Inorganic Sol-Gel Films", *in preparation.*

J. Yang, M.S. Oliver, R.H. Dauskardt, "Fatigue Debonding Characteristics of Fiber-Reinforced TiGr Laminates", *in preparation.*

CONFERENCE TALKS

October 2011, "Fracture in Hybrid Molecular Glass Films: Experiments and Computational Models," invited presentation at the International Materials Science and Technology Conference 2011, Columbus, OH.

October 2011, "Fatigue and Fracture in Thin-Film Devices and Hybrid Laminates," invited presentation at the International Materials Science and Technology Conference 2011, Columbus, OH.

September 2011, "Reliability of Hybrid Glass Films for Emerging Nanoscience and Energy Technologies," invited presentation at the ADMETA 2011 International Conference, Tokyo, Japan.

M.S. Oliver, G. Dubois, R.H. Dauskardt, "Molecular Structure and Design of Ultra-low-k Hybrid Glasses", Materials Research Society Spring Meeting, April 25-29, 2011

J. Yang, R.H. Dauskardt, "Hybrid Organic-Inorganic Glass Films for High-performance Adhesive Bonding", Materials Research Society Spring Meeting, April 25-29, 2011

J. Yang, R.H. Dauskardt, "High-Performance Organic-Inorganic Thin Film Structural Adhesive Interphases", TMS Annual Meeting & Exhibition, February 27-March 3, 2011.
May 2010, "Molecular Diffusion under Nanometer Scale Confinement during CMP of Nanoporous Films," invited presentation at the CMP Users Group meeting, Santa Clara, CA.

April 2010, "Molecular Modeling and Design of Hybrid Glasses," invited presentation at the 11th International Workshop on Stress-Induced Phenomena in Metallization, Dresden/Bad Schandau, Germany.

M.S. Oliver, G. Dubois, R.H. Dauskardt, "Molecular Design of Ultra-Low-k Hybrid Glasses", Proceedings of the IEEE International Interconnect Technology Conference, June 7-9, 2010

M.S. Oliver, R.H. Dauskardt, "Crack Growth Mechanisms in Sol-Gel Adhesive Coupling Layers", Proceedings of the International SAMPE Symposium, May 17-20, 2010

J. Yang, M.S. Oliver, R.H. Dauskardt, "Optimized Adhesive Bonding to Bulk Metallic Glass Substrates", Proceedings of the International SAMPE Symposium, May 17-20, 2010

M.S. Oliver, G. Dubois, R.H. Dauskardt, "Molecular Modeling and Design of Low-k Hybrid Glasses" Materials Research Society Spring Meeting, April 5-9, 2010

M.S. Oliver, R.H. Dauskardt, "Modeling the Molecular Network Structure of Hybrid Sol-Gel Materials", Materials Research Society Spring Meeting, April 5-9, 2010

J. Yang, M.S. Oliver, R.H. Dauskardt, "Optimized Adhesive Bonding Using Hybrid Organic-inorganic Sol-gel Films", Materials Research Society Spring Meeting, April 5-9, 2010

September 2009, "Modeling of Hybrid Glasses and Molecular Materials," invited presentation at the SAMPE 2009 International Conference, Wichita, KA.

M.S. Oliver and R.H. Dauskardt, "Simulation and Experiments of Fracture in Hybrid Organic-Inorganic Glasses", Materials Research Society Spring Meeting, April 13-17, 2009

R. H. Dauskardt, J. Yang and M. S. Oliver, “Optimized Adhesive Bonding to Bulk Metallic Glass Substrates”, TMS Annual Meeting & Exhibition, February 15-19, 2009.

M.S. Oliver and R.H. Dauskardt, “Hypersensitive Moisture-Assisted Crack Growth Along Sol-Gel Coupled Oxide/Epoxy Interfaces”, TMS Annual Meeting & Exhibition, February 15-19, 2009.

R. H. Dauskardt and M.S. Oliver, “Fatigue of TiGr Laminates,” invited presentation at the MS&T 2008 International Conference, October 2008.

M.S. Oliver, K.Y. Blohowiak, R.H. Dauskardt, “Reliability of Adhesive Interphases for Titanium Graphite Laminates”, SAMPE, May 18-22, 2008

M.S. Oliver, A. Kumar, K.Y. Blohowiak, R.H. Dauskardt, “High-Performance Metal/Epoxysilane Sol-gel Coupling Layers”, Materials Research Society Spring Meeting, March 24-28, 2008

R. H. Dauskardt, “Adhesion of Interfaces in Hierarchical Layered Structures for Emerging Technologies,” keynote presentation at the 137th Annual Meeting of the TMS, March 2008.

M.S. Oliver, K.Y. Blohowiak, R.H. Dauskardt, “Mechanisms of Delamination Growth in Titanium Graphite Hybrid Laminates”, Adhesion Society Annual Meeting, February 17-20, 2008

INTERACTIONS

Our research involves a strong collaboration with **Dr. Kay Blohowiak** and her colleagues at the Boeing Phantom Works, Seattle, WA. A new significant interaction involves **Dr. Steve Christensen** at Boeing related to molecular modeling of epoxy resin molecular networks. The interaction involves an exchange of computed networks from Boeing, which we use to predict mechanical and fracture properties. We have interacted with **Dr. Stacey Nyakana**, R&D Metallurgist at TIMET Corp., Henderson, NV, who has supplied us with Timetal 15-3 substrates and substrates with e-beam surface patterning are under consideration for study. The collaboration involves an exchange of laminate materials for study and resulting data. We have also been collaborating with **Dr. Geraud Dubois** at the IBM Almaden research center on sol-gel glass synthesis and computational modeling, and **Rich Moulton** at Applied Poleramics on the development of novel second phase toughened adhesives for high performance bonding. We have ongoing discussions with **Prof. Jonathan Stebbins**, Dept. of Geological and Environmental Sciences, Stanford, on high resolution NMR characterization of sol-gel interphase layers.

CONSULTATIVE FUNCTIONS

None

TRANSITIONS

None

AWARDS RECEIVED

Dauskardt received the following awards:

2011: The Henry Maso Award for fundamental contributions to the advancement of cosmetic and skin science, The International Federation of Societies of Cosmetic Chemists.

2011: The IBM Shared University Research Award in recognition of scientific and technological research achievements.

2010: The Semiconductor Industry Association University Researcher Award for research which has provided substantive and sustained contributions to semiconductor industry science and technology.

2010: The Metallurgical Society, Structural Materials Distinguished Scientist/Engineer Award for long lasting contributions to the fundamental understanding of microstructure, properties, and performance of structural materials for industrial applications, along with dedication and leadership of the Society.

2010: Elected Fellow of the ASM International for outstanding contributions to education and to the fields of mechanical behavior and fatigue of ceramics, metallic glasses, thin films and biomaterials.

2008: Elected Fellow of the American Ceramics Society.

2008: VLSI/ULSI Multilevel Interconnection (VMIC) International Conference Award for "Optimized Curing and CMP of Nanostructured Ultra-low-k Films," Fremont, CA.

2008: American Vacuum Society Thin Film User Group Special Award for contributions to the Northern California Chapter AVS, San Jose, CA.